Equivalent Thermal Conductivity of Epoxy Composites Filled with Aluminium and Red Mud Particles

A THESIS SUBMITTED
IN PARTIAL FULFILLMENT OF THE REQUIREMENT FOR THE DEGREE OF

Master of Technology

in

Mechanical Engineering
(Specialization: Thermal Engineering)

By

Johan Banjare
(Roll No. 212ME3308)

Department of Mechanical Engineering
National Institute of Technology
Rourkela, India
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C E RT I F I C A T E

This is to certify that the thesis entitled ***Equivalent Thermal Conductivity of Epoxy Composites Filled with Aluminium and Red Mud Particles***, submitted by **Johan Banjare** (Roll No: 212ME3308) has been carried out under my supervision in partial fulfilment of the requirements for the degree of ***Master of Technology*** *in* ***Mechanical Engineering (Specialization: Thermal Engineering)*** during session 2013 - 2014 in the Department of Mechanical Engineering, National Institute of Technology, Rourkela.

To the best of my knowledge, this work has not been submitted to any other University/Institute for the award of any degree or diploma.

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A B S T R A C T

The present research deals with the effect of volume fraction of filler particles on the effective thermal conductivity (k_{eff}) for polymer composites. This work sees an opportunity of enrichment of heat conduction ability of a typical particulate filled polymer composite. A mathematical correlation for the of polymer composites filled with elliptical shape particles is developed using the law of minimal thermal resistance and equal law of the specific equivalent thermal conductivity. To validate this mathematical model, two sets of epoxy based composites, with filler content ranging from 0 to 25 vol % have been prepared by simple hand lay-up technique. For first set, micro-sized aluminium particles are chosen as filler whereas for second set red mud is taken as a filler material whereas matrix material remains epoxy. Thermal conductivities of these composite samples are measured as per ASTM standard E-1530 by using the Unitherm™ Model 2022 tester. Experimentally measured values are then compared with the values obtained from the proposed mathematical model and also with models established earlier such as Rule-of-Mixture (ROM), Maxwell's model, and Bruggeman model. It is observed that the results obtained from the proposed model fits well with the experimental data. This study shows that the k_{eff} improves quite significantly as the conductive filler in the composite increases. An improvement of about 160 % in the value of thermal conductivity is recorded with addition of 25 vol % of aluminium filler in epoxy resin whereas when filler material is red mud it is noticed that k_{eff} increases by about 135 %. From this point of view a costly aluminium powder can be replaced by an industrial waste red mud powder as far as thermal conductivity is concerned. Some physical properties like density and void fraction together with morphological behaviour of the fabricated specimens are also reported in this investigation. With light weight and improved heat conductivity, these epoxy composites can possibly be used for applications such as heat sink, printed circuit boards, and encapsulation.

C O N T E N T S

CHAPTER 1

INTRODUCTION

1. BACKGROUND AND MOTIVATION

In electronic and electrical field micro-electronic packaging plays an important part. More amount of heat is generated when the electronic circuits work. This generated heat should be sufficiently dissipated to avoid thermal failure of packaging material, so that the thermal conductivity of packaging material must be high and also it have low value of CTE.

The heat dissipation in microelectronic packaging is becoming very serious problem. There are excellent electrical and thermal properties are found in Polymers. Though, thermal conductivity of common polymers, for example epoxy, polyester, polyethylene, polypropylene, and polyamide etc. has low thermal conductivities. Due to this reason, the polymers cannot efficiently dissipate heat when it is used in various works and thermal failure is occur due to their high CTE result in.

Under this circumstance too, emerged a class of promising packaging materials – polymer composites. Particulate filled polymer composites i.e. polymers filled with thermally conductive particulate matters are coming up as a cost effective way to cope with such thermal management issues.

COMPOSITE MATERIALS

There are basic two phase of composite material, in which one is known as matrix material and another one is called reinforcing material. The reinforcing material is embedded over matrix material. The matrix material is continuous phase and reinforcing is discontinuous phase. The reinforcing phase is much harder than matrix phase. In composite material matrix phase removes the stresses between reinforcing phase and also protect from mechanical and environmental damage. The function of reinforcing material is to improve mechanical and thermal properties of composites. Composite are hybrid of two or more material such as reinforced polymer, metal or ceramics. The reinforcement may be in the form of fibres, particles, whiskers or lamellae, and are incorporated in a suitable matrix, thereby providing a material that combines the most useful properties of the constituents. Generally the properties of composites are superior to those of its individual constituents. High structural strength, glass fibre reinforced plastics were developed in the early 1940's and the technology of reinforced polymers has progressed significantly since then. In a typical glass fibre reinforced plastic composite, the strength and stiffness are provided by the glass fibre while the temperature capabilities of the composites is governed by the plastic matric. These composites are finding increasing application in air craft, automobile industry.

Matrix material: - A variety of matrix polymer, metal, alloy, intermetallic, ceramics, carbon, cement etc. have been used for making composites. The matrix material serves several functions which are vital to the performance of the composite material. These functions also depend upon the type of reinforcement such as dispersions, particulates, whispers, discontinuous or continuous fibres. The matrix enables the composite to withstand compression, flexural and shear forces or tensile loads.The polymer material for a matrix can be thermosetting or thermoplastic resins. Thermoset resins, in the presence of a catalyst reaction or cure. Once cured these material can no longer flow. Thermosetting resins such as epoxies, bismaldeimides and cyanates can provide good strength properties. Thermoplastic resins are normally solids at room temperature but soften or melt when heated to elevated temperatures and become solid when cooled. Typical thermoplastic resins include polymide, polyami9de, polyphenylene sulphide (PPS), polyetherether ketone (PEEK), polyethylene terephthalate (PET), and polyetherulfone.

Reinforcement:- The reinforcement for composites may be in the form of particles, whiskers, fibres, lamellae or a mesh. They may increase the strength, stiffness or modify the failure mechanism advantageously. There can be special cases where the fibres may conduct or resist heat and electricity. Whiskers of metals, inter-metallic, oxides, carbides and nitrides are frequently used as reinforcement. A veriety of continuous fibres of glass, carbon Kevlar (aramid), silicon carbide, alumina, boron, tungsten etc. are used as reinforcement.

Types of Composite Materials

Composite materials are classified in following group:

(i) Metal Matrix Composites

(ii) Ceramic Matrix Composites

(iii) Polymer Matrix Composites

(a) Metal Matrix Composites:

In this type of composite material metal is used as matrix material. These composites have higher specific modulus, specific strength and better properties at higher temperatures and lower CTE as compare to other composites. These composites can be used in chamber nozzle (in rocket, space shuttle), housings, tubing, and structural members etc. Metal matrix composite made a breakthrough in the development of useful properties of metals and alloys in relation to the traditional approach of alloying and heat treatment. These composites, containing discontinuous or continuous reinforcement with particulates, whiskers or fibres are capable of providing properties not achievable in monolithic alloys. Ceramic particles or whiskers dispersed in a metal matrix either by powder metallurgy or molten metal processing can enhance the modulus, strength; wear resistance, elevated temperature properties or control of thermal conductivity or coefficient of thermal expansion. The improved thermal conductivity and controlled coefficient of thermal expansion of SiC particle reinforced aluminum matrix composites are finding newer application as electronics packaging materials. Many potential aerospace applications have been identified for MMC. These are also having automotive applications.

(b) Ceramic matrix Composites:

Ceramics are characterized by lightness, hardness, corrosion and oxidation resistance and superior elevated temperature properties. Hence many ceramic matrix composites can be used as temperatures higher than those of the polymer and metal matrix composites. Important ceramic matrixes are oxides, carbides, nitrides, borides, glasses, glass-ceramics and silicates. The reinforcements used are SiC, Si3N4, Al2O3, BN, ZrO2, AIN and C in the form of fibers, whiskers or particulates. These composites are processed by sintering, hot pressing, hot isotactic pressing, infiltration, reaction bonding and combustion synthesis. Currently ceramic matrix composites are used as cutting tool inserts wear resistant composites, space shuttle tiles and aerospace components. Other potential application includes engine components, armor of military vehicles, and leading edge application in aerospace and high temperature corrosion resistant parts. Other potential application includes bio-ceramic and high temperature ceramic super conductor composite wires for power transmission cables, motors and super conducting magnetic energy storage system.

(c) Polymer Matrix Composites:

In these type of composite material polymer is used as matrix material. Now days common used matrix materials are polymeric. The mechanical properties of polymers are insufficient and it has low strength and stiffness is compared to other composites. Through the adding other reinforcing material with polymer the above difficulties can be overcomes. The procedure of preparing polymer composites is much easier than other composites; it doesn't require any high pressure and high temperature operation. By the use of simple hand lay-up technique it can be manufactured. Due to this reason polymer composites were established quickly and shortly became popular for structural applications. It can be used in electronic field, heat sink, circuit board etc. In case of the reinforced plastics, the characteristics of the desired end product such as size, shape, function and quality determine the method by which the basic materials are combined, moulded, cured and machined. Prominent moulding process for reinforced plastics includes hand lay-up, spray up moulding, prepare lay-up (vaccume bag and auto-clave moulding), press moulding (SMC, BMC etc.), resin injection moulding and filament winding. To make useful structure, the composite materials have to be joined and machined. The industrial applications of reinforced plastics have spread a wide spectrum of consumer goods, constructions, chemical plants, marine and road transportation and aerospace components. Mechanical engineering products include cylinders, rolls, shafts, coupling, spindles, robot arms, covering etc. the aerospace industry used a wide range of products including floor panels, skin panels, elevators, wings, flaps, covers, tanks, struts and rotor blades.

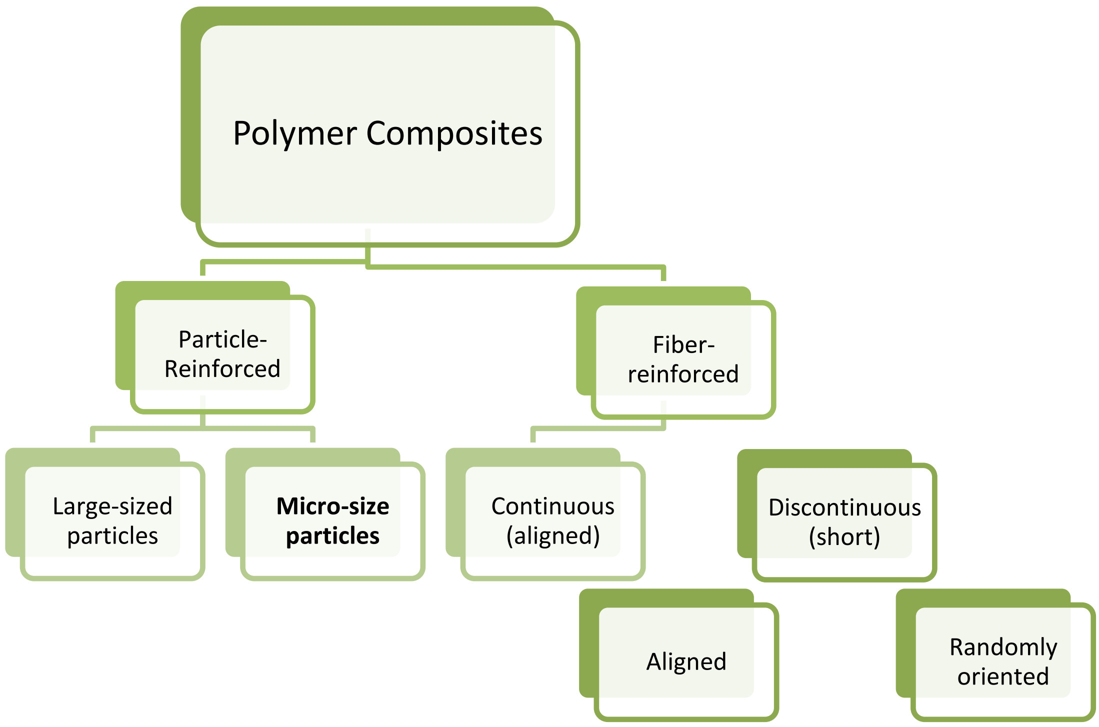

Figure 1.1 Classification of composites based on reinforcement type

Polymer composites are classified into two basic categories:

- Particle reinforced polymer
- Fiber reinforced polymer

Fiber reinforced polymer

It is also known as fibre-reinforced plastic. In this type of polymer composite fibres are reinforced with a polymer matrix. The common used fibres are glass, carbon, or aramid, while other fibres such as paper or wood or asbestos can also be used. The Fibre reinforced polymer composites can potentially application in the aerospace, automotive, marine, and construction industries.

Particle reinforced polymer:

Particulate composites have an additive constituent which is essentially one or two dimensionally and macroscopic /microscopic. In some composites however, the additive constituent is macroscopically non-dimensionally, i.e., conceptually a point, as opposed to a line or a area. Only on the microscopic scales does it become dimensional, i.e., a particle, and thus the concept of composite must come down to the microscopic level if it is to encompass all the composite of interest of engineers. Particulate composites differ from the fibre and flake types in that distribution of the additive constituent is usually random rather than controlled. Particulate composite are therefore usually isotropic. This family of composites includes dispersion-hardened alloy and cermet.

1.2 INTRODUCTION TO THE RESEARCH WORK

The present research is essentially an experimental investigation focused on the thermal characteristics of a new class of particulate filled polymer composites. It also includes fabrication of the composites and their physical and mechanical characterization.

Shrinking of electronic devices due to their less space and higher portability is the demand of the customer of recent times. Applications in electronic industry therefore require superior materials for packaging and encapsulation. A modern packaging application therefore embodies a host of materials and necessitates an integrated design approach from the very outset.

Last few decades have seen a rapid advancement in electronics technology as there is a constant demand for miniaturization and hence, the sizes are gradually shrinking whereas the number of components and communication speed are increasing day-by-day which leads to generation of high amount of heat and gives rise to problems of heat dissipation. Neat polymers like epoxy or polyester are commonly used as materials for heat sink applications but they suffer from a drawback of low thermal conductivity. If these polymers are filled with metal powders like aluminum or copper, the thermal conductivity improves. Therefore, in order to enhance the thermal conductivity, thermally conductive fillers of suitable volume fraction can possibly be used in polymers for such applications. This concept has been imbibed as the very basis of the present research.

Thermal conductivity is an important parameter for thermal characterization of inorganic-particulate filled polymer composites. Various complicated factors affect the thermal conductivity of such materials. In view of this, in the present research, theoretical heat transfer models for estimation of effective thermal conductivity of filled polymer (elliptical filler) are developed based on the law of minimal thermal resistance and equal law of specific equivalent thermal conductivity.

Epoxy is chosen as the polymer to be used as the matrix material for this work. Thus speciality epoxy resins and curing agent designed for use in high performance composites have excellent elevated temperature resistance, good mechanical properties, low water absorption and relatively high glass transition temperature. Hence a proper combination of epoxy resin, modifying resin, reinforcing fibre, curing agent and processing procedure are required to take full advantages of their properties. Since the relatively low strength polymers can be converted into high strength composites by reinforcement, this field of the composite industry is well established and is growing faster into newer application.

For first set of polymer composite micro- sized aluminium powder is used as filler material. It is a silvery white metal. Aluminium is considered to be remarkable because of its low density and its ability to resist corrosion. Aluminium is primarily chosen because it is highly conducting in nature. For second set of composite, an industrial waste red mud powder is used as filler material. Red mud is the insoluble product generated after the digestion of bauxite with alkali sodium hydroxide at high temperature and pressure to produce alumina. This process of extraction of alumina is known as Bayer’s process. The red mud is used as clay material for building application and some time it is used for pollution control (treatment of waste water).

1.3 THESIS OUTLINE

The rest of this thesis is ordered as follows:

Chapter 2: It gives a literature review. This part of thesis is reports the research works of earlier investigators on thermal characterization of particulate reinforced polymer composites.

Chapter 3: Presents the development of mathematical model for evaluation of effective thermal conductivity of elliptical particles filled polymer composites.

Chapter 4: This part of thesis gives description of the raw materials and the method used for their test procedures. It presents the details of fabrication of composite specimens and characterization of the composites under investigation.

Chapter 5: It gives the result obtain from theoretical calculations and experimental works of fabricated composites. Some physical results are also given in this chapter.

Chapter 6: It gives the conclusion of research work on the basis of obtained results and also gives some ideas for future scope.

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- On particulate filled polymer composites
- On thermal characteristics of particulate filled polymer composites
- On metal powder as a filler materials
- On aluminium powder as a filler materials
- On industrial waste as a filler materials
- On red mud as a filler materials
- On thermal conductivity models for particulate filled polymer composites

2.1 ON PARTICULATE FILLED POLYMER COMPOSITES

The shape, size, volume fraction and specific surface area of such added particles have been found to affect mechanical properties of the composites greatly. Yamamoto et al. [1] told that the structure and shape of silica particles have significant effects on the mechanical properties such as fatigue resistance, tensile and fracture properties. Moloney et al. [2] and Adachi et al. [3] give that the mechanical properties of epoxy composites were dependent on volume fraction of particles. Furthermore, effects of different particles of micron size magnitude and nano-particles on the properties of the composites were given by Yuan et al. [4]. As per the filler theory [5], optimal composite properties are achieved when the particle size distribution permits a maximal packing of the filler particles and according to the mastic theory , the matrix creates a coating on each filler particle with an optimal thickness. The fillers also affect the tensile properties according to their packing characteristics, size and interfacial bonding. The maximum volumetric packing fraction of filler reflects the size distribution and shape of the particles [6].

2.2 ON THERMAL CHARACTERISTICS OF PARTICULATE FILLED POLYMER COMPOSITES

Carbon-based fillers with high thermal conductivity and low density appear to be the most promising fillers. Graphite, carbon fiber and carbon black are well-known carbon-based fillers. Graphite is considered as the best conductive filler because of its good thermal conductivity and low cost [7]. Graphite with single graphene sheet shows intrinsically high thermal conductivity of about 800W/m-K [8]. Expanded graphite, an exfoliated form of graphite with layers of 20-100nm thickness, has also been used in polymer composites [9]. It was found that thermal conductivity of the chemically functionalized graphite/epoxy composite with such exfoliated graphite (20wt %) increased from 0.2 to 5.8 W/m-K. Carbon fiber, typically vapor grown carbon fiber (VGCF) is important carbon-based filler [10]. Studies conducted on modified thermal conductivity of polymer composites filled with carbon nanotubes have recently been reviewed by Han and Fina [11].

Ceramic powder reinforced polymer materials have been used extensively for such applications because of their high thermal and low electrical conductivity. Some promising ceramic fillers such as SiC [12] are in use to improve thermal conductivity of various polymers. It is observed from the above literature on ceramic particle filled polymers that there is an appreciable increase in thermal conductivity of the composite with increase in filler concentration whereas no significant changes are observed in electrical conductivity of such composites.

Kumlutas and Tavman [13] have developed a numerical model for particulate filled polymers which shows good agreement with the experimental values.

Apart from heat dissipation problem, high coefficient of thermal expansion (CTE) of matrix material is also a major source of thermal failure in many applications where material is subjected to periodic heating and cooling due to thermal cycling. Iyer et al. [14] have recently reported significant reduction in CTE as the content of boron nitride is increased in the composite. Dey et al. [15] have studied the dependence of CTE on volume fraction of filler at ambient temperature. Yasmin et al. [16] have reported that, as the graphite concentration in epoxy increases to about 2.5 wt %, glass transition temperature increases and CTE of the composite decreases, however on further addition of graphite i.e. 5 wt%, the CTE of the composite starts increasing. Thomas et al. [17] studied about the effect of $Sm_2Si_2O_7$ particles as reinforcement in two different matrix materials (polyethylene and polystyrene) on CTE.

2.3 ON METAL POWDER AS A FILLER MATERIALS

Sofian et al. [18] studied the effect of various metal powders like copper, zinc, iron and bronze on the thermal properties like conductivity, diffusivity and specific heat of high-density polyethylene matrix. Mamunya et al. [19] later reported the improvement in thermal conductivity of two different categories of polymers i.e. thermoplastic (polyvinyl chloride) and thermoset (epoxy) filled with copper and nickel particles. Though in 1990s, Tecke et al. [20] and Tavman [21] had already used copper powder as filler and measured the thermal conductivity of the composites by hot disk method. They have reported around 150% increase in the value of thermal conductivity for the composites filled with 24 vol% of copper. Silver too has high potential to be used as filler because of its high thermal conductivity. The effect of silver particulates in epoxy was studied by Bjorneklett et al. [22]. The filling of a polymer with metallic particles though resulted in increased thermal conductivity; simultaneous increase in density of the composites was also recorded, thus restricting the use of metal powders for light-weight applications. The method of incorporation of preformed gold nanoparticles (AuNPs) into the acrylic polymer (AP) matrices and optical, TEM characterization of AuNP/AP bulk and film composite was reported by Burunkova et al. [23]. Gold nanoparticles - polyaniline, graphene sheets - polyaniline composite materials were fabricated and their structure and electrical properties were analyzed by Gorshkov et al. [24]. The morphology and structure of the monodispherse gold microspheres with novel hierarchical structure and their composite film are characterized, and its catalytic effect on reduction of p-nitrophenol was investigated by Xia et al. [25].

2.4 ON ALUMINIUM POWDER AS A FILLER MATERIALS

Tavman [26] took aluminium powders as filler and studied the thermal property of high density polyethylene whereas later Boudenne et al. [27] gave an overview on the thermal conductivity of polypropylene/aluminium composites. Morphology and thermal conductivity of polyacrylate composites with various fillers such as multi-walled carbon nanotube (CNT), aluminum flake (Al-flake), aluminum powders and Al–CNT was reported by Choi et al. [28]. Yield and internal stresses in aluminum filled epoxy resin with influence of the filler content was investigated by Goyanes et al. [29]. Carson et al. [30] studied the effective diffusivity of linear-medium-density-polyethylene/aluminium composites was measured for a range of volume fractions using a simple, transient comparative method and effective thermal conductivity data were calculated from the effective thermal diffusivity data. The thermal conductivity of polymer composites having a matrix polystyrene (PS) containing aluminium nitride (AlN) reinforced was reported by Yu et al. [31].

2.5 ON INDUSTRIAL WASTE AS A FILLER MATERIALS

Industrialization is must for uplifting nation's economy in developing countries, it has also caused the generation of significant quantities of solid wastes that lead to serious problems relating to environmental pollution. Therefore, wastes seem to be a byproduct of growth. But a country like India can ill afford to lose them as sheer. Production of industrial slag dates back to the beginning of extracting of metals from ores through metallurgical processes. Copper slag is such a by-product obtained during the matte smelting and refining of copper reported by Biswas et al. [32]. Shi et al. [33] have reported a detailed review on utilization of copper slag in the manufacturing of cement and concrete.

A new kind of cement made of clinker, steel slag, fly ash, and certain admixtures has been developed by Xuequan et al. [34], Cheng and Chiu [35] researched the usage of granulated blast furnace slag as a filler material in making of geo-polymers.

2.6 ON RED MUD AS A FILLER MATERIALS

Red mud as a partial substitute of clay in ceramic products like bricks, tiles etc. and as an additive for mortar and concrete [36]. Use of red mud in agricultural applications such as in acidic soils or as a treatment for iron deficient soils has also been reported [37]. Red mud finds some applications in ceramic industries as well. Yalcin et al. Satapathy et al. [38] have reported the coating potential of red mud for deposition on various metal substrates using plasma spray technology. Numerous other uses for red mud have been reported and well documented by Thakur and Das [39]. Erosion characteristics of red mud filled bamboo–epoxy and glass–epoxy composites was reported by Biswas et al. [40], The morphological study of Poly (vinyl alcohol)-Modified red Mud Composite Materials was reported by Bhat et al. [41], addition of red mud in to sisal fiber, banana fiber reinforced unsaturated polyester (USP) was investigated by Prabu et al. [42].

2.7 ON THERMAL CONDUCTIVITY MODELS FOR PARTICULATE FILLED POLYMER COMPOSITES

Numerous theoretical and empirical models have been proposed in the past to estimate and predict the effective thermal conductivities of particulate filled composites.

For **parallel conduction** model [43]

$$k_c = \left(1-\phi_1-\phi_2\right)k_m + \phi_1 k_{f_1} + \phi_2 k_{f_2} \tag{2.1}$$

where, k_{f1}, k_{f2}, k_m, k_c are thermal conductivities of 1st filler, 2nd filler, composite matrix, conductivity of the composite as a whole and ϕ_1 and ϕ_2 are volume fractions of 1st and 2nd filler respectively.

For **series conduction** model [43]

$$\frac{1}{k_c} = \frac{1-\phi_1-\phi_2}{k_m} + \frac{1}{k_{f_1}} + \frac{1}{k_{f_2}} \tag{2.2}$$

The correlations represented by Equations (2.1) and (2.2) are derived on the basis of the rules-of-mixture.

The **geometric mean model** [44], also known as **Ratcliffe Empirical Model** gives the effective thermal conductivity as:

$$k_c = k_m^{(1-\phi_1-\phi_2)} k_{f_1}^{\phi_1} k_{f_2}^{\phi_2} \tag{2.3}$$

Bruggeman [45] derived an equation employing different assumptions for permeability and field strength for dilute suspension of spheres for a homogeneous medium and the implicit equation is given as:

$$1-\phi=\left[\frac{k_c-k_f}{k_m-k_f}\right]\left(\frac{k_m}{k_c}\right)^{1/3} \tag{2.4}$$

Maxwell [46] has obtained an exact expression for thermal conductivity, using potential theory for an infinitely dilute composite of spherical particulates dispersed randomly and devoid of mutual interaction in a homogeneous medium, which is given by

$$k_c=k_m\left[\frac{k_f+2k_m+2\phi\left(k_f-k_m\right)}{k_f+2k_m-2\phi\left(k_f-k_m\right)}\right] \tag{2.5}$$

where k_c ,k_m and k_f are thermal conductivities of composite, continuous- phase (matrix), and dispersed-phase (filler) respectively, and ϕ is the volume fraction of the dispersed-phase. Eq. (3.5) is well known for dilute composites which is the earliest flux law in which a cube of suspension for a single particle was considered.

Lewis and Nielsen [47] derived a semi-theoretical model by modification of the Halpin-Tsai equation for a two phase system which assumes an isotropic particulate reinforcement and also takes into consideration the shape of particle as well as its orientation.

$$k_c=k_m\left[\frac{1+AB\phi}{1-B\phi\psi}\right] \tag{2.6}$$

Where $B=\left[\frac{\left(k_f/k_m\right)-1}{\left(k_f/k_m\right)+A}\right] and, \psi=1+\left[\frac{1-\phi_m}{\phi_m^2}\right]$

where, k_f is thermal conductivity of filler material and ‘ϕ’ is the volume fraction of filler material.

2.8 KNOWLEDGE GAP IN PREVIOUS INVESTIGATIONS

From above literature it can be seen that, there is some knowledge gap that demands a well-organized and systematic research in this area of particulate filled polymer composites. A comprehensive review of the available literature tells that:

1. There is no report available on the development of the theoretical model for evaluating k_{eff} for elliptical shaped particulate filled polymer composites.

2. There is no work available on any industrial waste to be used for enhancing the thermal conductivity of polymers.

3. From the above literatures it can be seen that there is no model which can predict the k_{eff} with considered the combined effect of filler type, size, shape and distribution.

4. There is no work reported on the replacement of costly metal by any industrial waste.

2.9 OBJECTIVES OF THE PRESENT WORK

1. Development of mathematical models for estimation of k_{eff} of elliptical shape particulate filled polymer composites.

2. Fabrication of composites using micro-sized aluminium powder as the reinforcing filler with an objective to enhance the k_{eff} of neat polymer.

3. Fabrication of a new class of composites using industrial waste i.e. micro-sized red mud powder as the reinforcing filler with same objective.

4. To study the morphological behavior of these fabricated composite.

5. Theoretical and experimental analysis of density of fabricated composites.

6. Measurement of k_{eff} of all the above fabricated composites.

7. Validation of the values obtained from mathematical model by comparing the k_{eff} values obtained experimentally for both sets of fabricated composites.

Chapter Summary

This chapter has provided

- Back ground information of polymer composites.
- Knowledge gap in previous investigation.
- The objectives of the present work.

The next chapter gives the development of mathematical model for evaluating k_{eff} of elliptical shaped particulate filled polymer composites.

****

CHAPTER 4

MATERIALS AND METHODS

4.1 MATERIALS

4.1.1 Matrix Material

Matrix materials are of different types like metals, ceramics and polymers. Polymer matrices are most commonly used because of cost efficiency, ease of fabricating complex parts with less tooling cost and they also have excellent room temperature properties when compared to metals and ceramic matrices. Polymer matrices can be either thermoplastic or thermoset. Thermoset matrices are formed due to an irreversible chemical transformation of the resin into an amorphous cross-linked polymer matrix. Due to huge molecular structures, thermoset resins provide good electrical and thermal insulation. They have low viscosity, which allow proper fiber wet out, excellent thermal stability and better creep resistance.

The most commonly used thermoset resins are epoxy, polyester, vinyl ester and phenolics. Among them, the epoxy resins are being widely used for many advanced composites due to their excellent adhesion to a wide variety of fibers, superior mechanical and electrical properties and good performance at elevated temperatures. In addition to that they have low shrinkage upon curing and good chemical resistance. Due to several advantages over other thermoset polymers as mentioned above, epoxy (LY 556) is chosen as the matrix material for the present research work. It chemically belongs to the 'epoxide' family. Its common name is Bisphenol-A-Diglycidyl-Ether (commonly abbreviated to DGEBA or BADGE) and its molecular chain structure is shown in Figure 4.1. It provides a solvent free room temperature curing system when it is combined with the hardener tri-ethylene-tetramine (TETA) which is an aliphatic primary amine with commercial designation HY 951 (Figure 4.2). The LY 556 epoxy resin (Figure 4.3) and the corresponding hardener HY-951 are procured from Ciba Geigy India Ltd. Table 4.1 provides some of the important properties of epoxy.

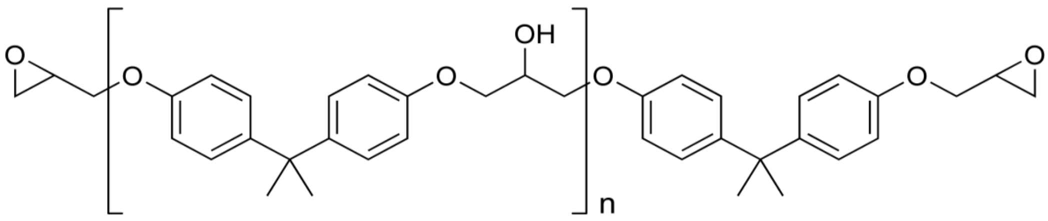

Figure 4.1 Basic epoxy resin chain (‘**n**’ denotes number of polymerized unit)

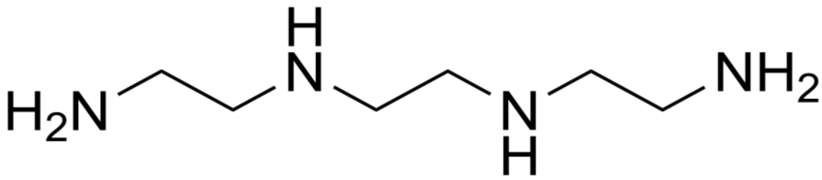

Figure 4.2 Tri-ethylene-tetramine (hardener used for epoxy matrix)

Figure 4.3 Epoxy resin and hardener

Table 4.1 Some important properties of epoxy

Characteristic Property	Inferences
Density	1.1 gm/cc
Compressive strength	90 MPa
Tensile strength	58 MPa
Thermal conductivity	0.363 W/m-K
Coefficient of Thermal expansion	62.83 ppm /°C

4.1.2 Filler Material for first set of polymer composite (aluminium powder)

For first set of polymer composite micro- sized aluminium powder is used as filler material. It is a silvery white metal. Aluminium is considered to be remarkable because of its low density (2.7gm/cm3) and its ability to resist corrosion. Aluminium is primarily chosen because it is highly conducting in nature possesses thermal conductivity of around 205 W/m-K. Structural components made from aluminium and its alloys are vital to the aerospace industry and are very important in other areas of transportation and building. The pictorial view of micro-sized aluminium powder used in this work is given in (Figure 4.4). Table 4.2 shows some important properties of aluminium powder.

Figure 4.4 Micro sized aluminium powder used as filler in the present work

Table 4.2 Some important properties of aluminium powder

Characteristic Property	Inferences
Density	2.7 gm/cc
Compressive strength	150 MPa
Tensile strength	325 MPa
Thermal conductivity	205 W/m-K

4.1.3 Filler Material for second set of polymer composite (red mud powder)

For second set of polymer composite an industrial waste red mud powder is used as filler material. Red mud is the insoluble product generated after the digestion of bauxite with alkali sodium hydroxide at high temperature and pressure to produce alumina. This process of extraction of alumina is known as Bayer's process. (Figure 4.5) shows the flow chart of red mud generation.

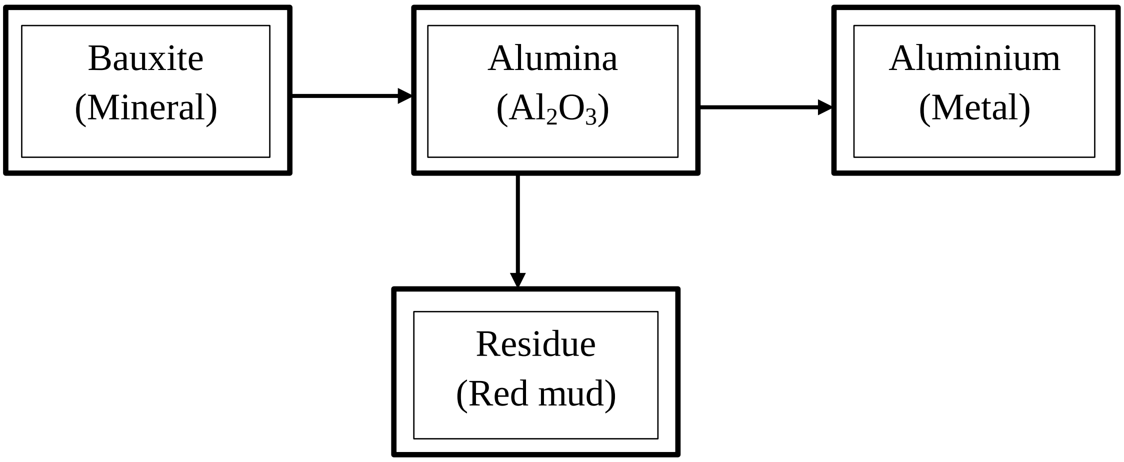

Figure 4.5 Flow chart of Bayer's process.

It has wide range of application starting from in the field of building (clay material, cements, ceramics, fired and non-fired building materials, concrete industry), pollution control (treatment of waste water and polluted waste gases), metal recovery (iron, titanium, aluminum, alkali), coagulant, adsorbent, catalyst and in soil remediation. Its thermal conductivity and density values are 11.7 W/m-K and 3.1gm /cm3 respectively, which make this material suitable for present application. Figure 4.6 shows the pictorial view of micro-sized red mud powder used in this work. Table 4.3 shows some important properties of red mud powder.

Table 4.3 Some important properties of red mud powder

Characteristic Property	Inferences
Density	3.1 gm/cm^3
Compressive strength	50 MPa
Tensile strength	75 MPa
Thermal conductivity	11.7 W/m-K

Figure 4.6 Micro-sized red mud powder used as filler in epoxy

4.2 EXPERIMENTAL DETAILS

4.2.1 Composite Fabrication

Epoxy Composites filled with micro-sized aluminium powder

Conventional hand lay-up technique is used for preparation of polymer composite specimens. For first set of polymer composite micro-sized aluminium particles are reinforced in epoxy resin. Epoxy resin and the corresponding hardener are mixed in a ratio of 10:1 by weight as per recommendation. Dough is prepared by mixing micro-sized aluminium powder with epoxy resin. A paper cup is coated with uniform thin film of silicone-releasing agent, then dough is slowly poured into that molds. After that the castings are left to cure at room temperature for about 24 hours after which the samples are released. Composites of five different compositions with 5vol%, 10vol%, 15vol%, 20vol% and 25vol% of fillers are prepared. These released composites are disc-type specimens (diameter 50 mm, thickness 3 mm). The composition of aluminium/epoxy fabricated composite is shown in table 4.4 and (figure 4.8) shows fabricated aluminium epoxy composite.

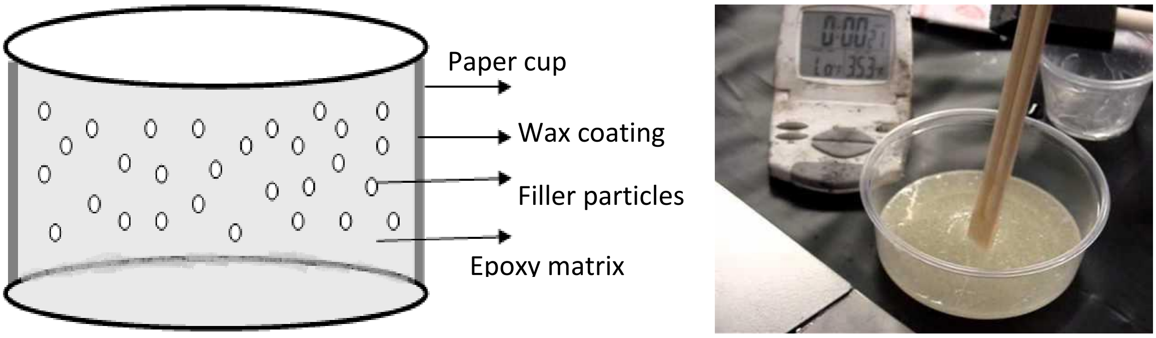

Figure 4.7 Particulate filled epoxy composite fabrication by hand lay-up process

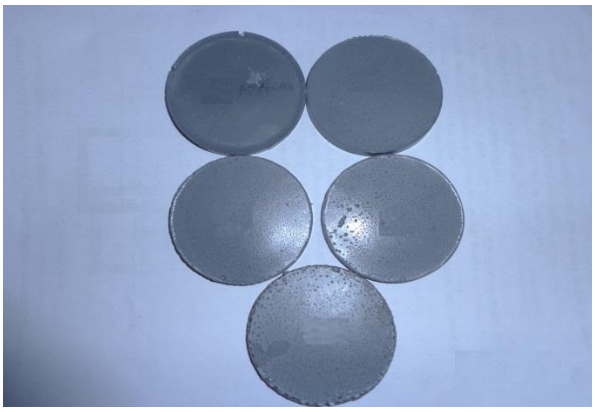

Figure 4.8 Micro sized aluminium filled epoxy composites

Table 4.4 Epoxy composites filled with micro-sized aluminium particles (**Set 1**)

Sample no.	Composition
1	Epoxy + 5 vol % Aluminium particle
2	Epoxy + 10 vol % Aluminium particle
3	Epoxy + 15 vol % Aluminium particle
4	Epoxy + 20 vol % Aluminium particle
5	Epoxy + 25 vol % Aluminium particle

Epoxy Composites filled with micro-sized red mud powder

Using the same hand lay-up technique, micro-sized red mud particles were reinforced in the epoxy resin to prepare the second set of polymer composites in different proportions (Table 4.5) according to the experimental need. (Figure 4.9) shows fabricated red mud filled epoxy composite.

Table 4.5 Epoxy composites filled with micro-sized red mud particles (**Set 2**)

Sample no.	Composition
1	Epoxy + 5 vol % Red mud particle
2	Epoxy + 10 vol % Red mud particle
3	Epoxy + 15 vol % Red mud particle
4	Epoxy + 20 vol % Red mud particle
5	Epoxy + 25 vol % Red mud particle

4.3 CHARACTERIZATION

4.3.1 Scanning electron microscopy

Scanning Electron Microscope (SEM) JEOL JSM-6480LV is used to study the shape and size of the filler particles and the distribution characteristics of such filler particles into the matrix body. The study is done for both aluminium/epoxy and red mud/epoxy composites. All the composite samples are mounted on stubs with silver paste. Before the micrographs are taken, a thin film of platinum is vacuum-evaporated on composites, for enhance the thermal conductivity of the samples.

4.3.2 Density Determination

The densities of all the fabricated composites are measured by well-known instrument Pycnometer. Density determination by pycnometer is considered to be very precise method. Pycnometer works on Archimedes principle. According to Archimedes principle, when an object is immersed in a liquid the apparent loss of weight of an object is equal to the upthrust and this is also equal to the weight of the liquid displaced.

It uses a working liquid with known density, such as water. Pycnometer can used to determine the density of homogeneous solid object that does not dissolve in working liquid. First the pycnometer is filled with distilled water.

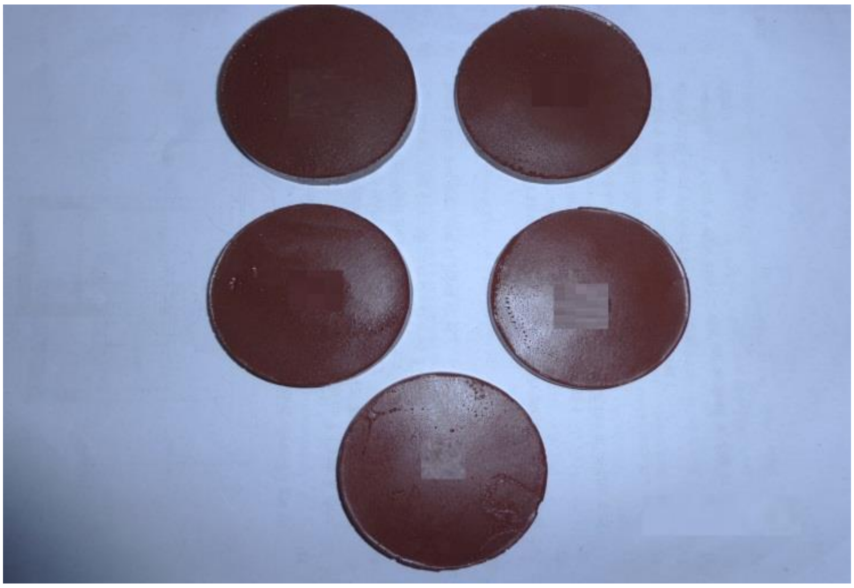

Figure 4.9 Micro-sized red mud filled epoxy composites

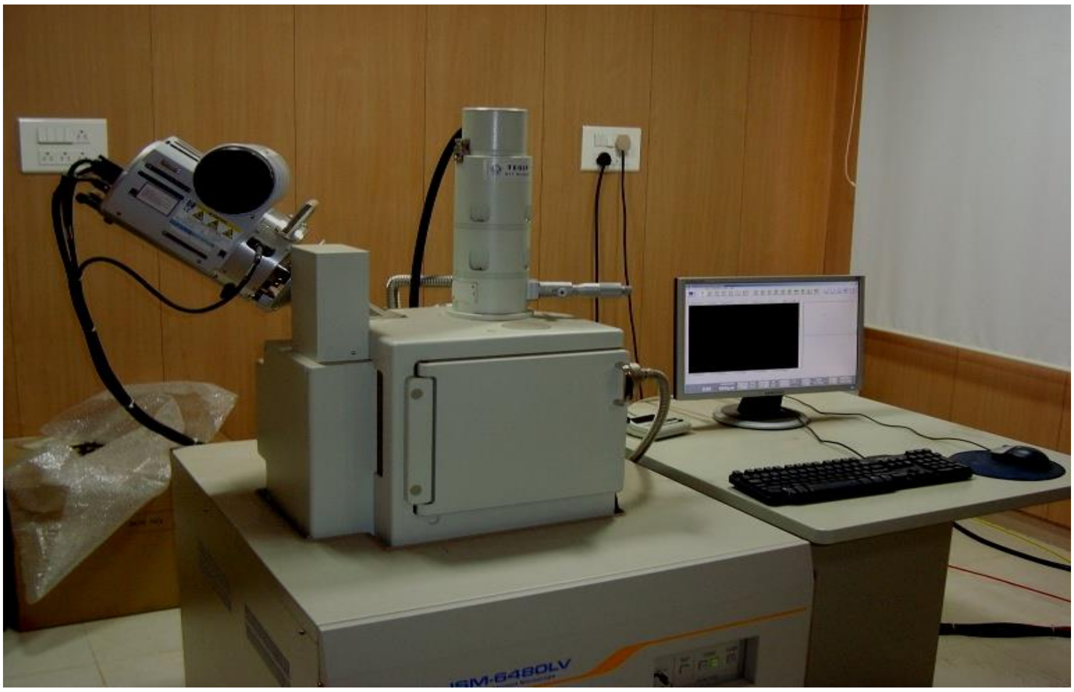

Figure 4.10 Scanning Electron Microscope (JEOL JSM-6480LV)

Volume of water filled in pycnometer and the stopper is given by

$$V = \frac{m_{H_2O}}{\rho_{H_2O}} \tag{4.1}$$

where m_{H2O} is experimentally measured weight of water (pycnometer weight subtracted). That the pycnometer is dried and weight of pycnometer together with inserted composite specimen (m_c+m_s) is taken. Than weight m'_{H2O} [(measured weight)-(m_c+m_s)] is determined by adding water into it. The volume of added water V'_{H2O} can be obtained as:

$$V'_{H_2O} = \frac{m'_{H_2O}}{\rho_{H_2O}} \tag{4.2}$$

The volume of solid composite "V_c" is the difference between the volume of water that fills the empty pycnometer V and volume V'_{H2O}

$$V_c = V - V'_{H_2O} = \frac{m_{H_2O} - m'_{H_2O}}{\rho_{H_2O}} \tag{4.3}$$

Finally density ρ_c can be then calculated as

$$\rho_c = \frac{m_c}{V_c} \tag{4.4}$$

Where mc = Mass of fabricated composite

Vc = Volume of fabricated composite

ρ_c = Density of fabricated composite

4.3.3 Thermal conductivity: experimental determination

Unitherm™ Model 2022 Thermal Conductivity Tester was used to measure the thermal conductivity of various materials, which include polymers, glasses, ceramics, rubbers, composites, few metals and other materials with medium to low thermal conductivity. Fluid or semi-fluids like paste etc. can be tested using a special container. In the present work, this instrument is used to measure the room temperature effective thermal conductivity of the composite specimens. Disc type specimens are used for this purpose. This test is conducted in accordance with ASTM E-1530 standards. The pictorial view of the Unitherm™ Model 2022 tester is given in Figure 4.11.

Figure 4.11 Thermal Conductivity Tester Unitherm™ 2022

By definition "Thermal conductivity is the exchange of energy between adjacent molecules and electrons in a conducting medium, it is a material property that describes heat flow within a body for a given temperature difference per unit area."
For one-dimension heat flow, the equation is given as:

$$Q = \kappa A \frac{T_1 - T_2}{x} \qquad (4.5)$$

where, Q is the heat flux (W), A is the cross-sectional area (m^2), k is thermal conductivity (W/m-K), x is the sample thickness (m), $T_1 - T_2$ is the temperature difference between surfaces (°C or K).

The thermal resistance of the sample is given as:

$$R = \frac{T_1 - T_2}{QA} \tag{4.6}$$

where, *R* is sample resistance between hot and cold surfaces. (m^2-K / W)
From the former equation, we can write

$$k = \frac{x}{R} \tag{4.7}$$

In Unitherm™ 2022, transducers measure the value of heat flux *Q* and temperature difference between upper and lower plate. Thus, thermal resistance between surfaces can be evaluated. Providing different thickness and known cross-sectional area as input parameters, the sample thermal conductivity can be calculated.

Chapter Summary

This chapter provided

- Information of material used for present research work.
- Fabrication process of composite specimens.
- Physical, micro-structural and thermal characterization of fabricated composite specimens.

The next chapter presents the results result obtain from theoretical calculations and experimental works of fabricated composites. Some physical result is also given in the next chapter.

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CHAPTER 5

RESULTS AND DISCUSSION

5.1 MORPHOLOGY OF FABRICATED COMPOSITE SPECIMENS

The shape and size of the pure fillers i.e. aluminium powder and red mud powder are shown in Figure 5.1and 5.2 respectively. It can be seen that the shape of both filler particles i.e. aluminium and red mud are more or less elliptical in shape.

The distribution morphology of fabricated composites i.e. aluminium/epoxy and red mud/ epoxy are shown in Figure 5.3(a) and 5.3(b) respectively. In both sets of composites, filler content is of 20 vol%. From both the figures it can be clearly seen that the distributions of filler particles in epoxy resin for the fabricated specimens are almost uniform. Beyond 20% volume fraction of filler content it is quite difficult task to increase filler loading into epoxy resin. With the increases of filler loading the inter particle distance reduces continuously up to limit that particle start to interfere to each other. For both aluminium and red mud filled composites, it is observed that the both filler particles occupy the space between the epoxy matrixes, resulting in high packing density of fillers in matrix, and thus heat conductive networks are easily formed in epoxy matrix.

5.2 DENSITY OF FABRICATED COMPOSITE SPECIMENS

Density is a material property which is of prime importance in several weight sensitive applications. Thus, in many such applications polymer composites are found to replace conventional metals and materials primarily for their low densities. The density of a composite depends on the relative proportion of matrix and the reinforcing materials. In the present work, the densities of both the fillers are higher than the pure epoxy. Pycnometer is used to measure the density of fabricated sets of composites experimentally.

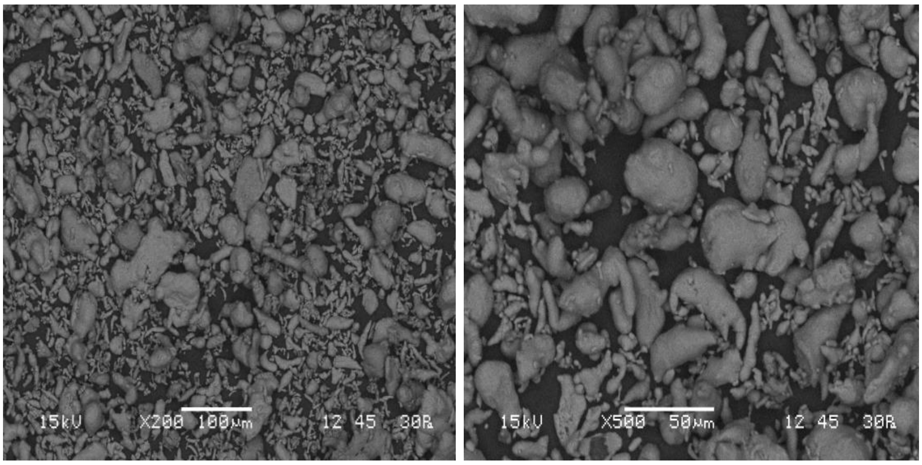


Figure 5.1 SEM images of pure aluminium powder

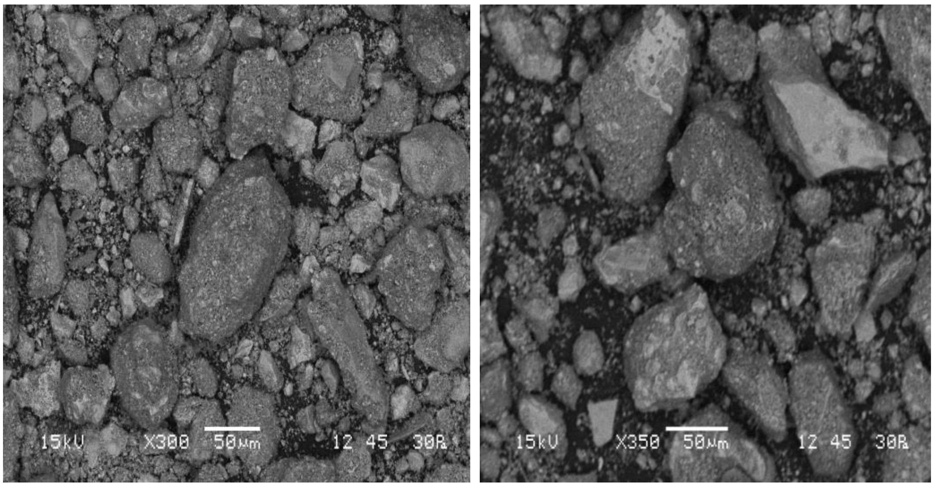
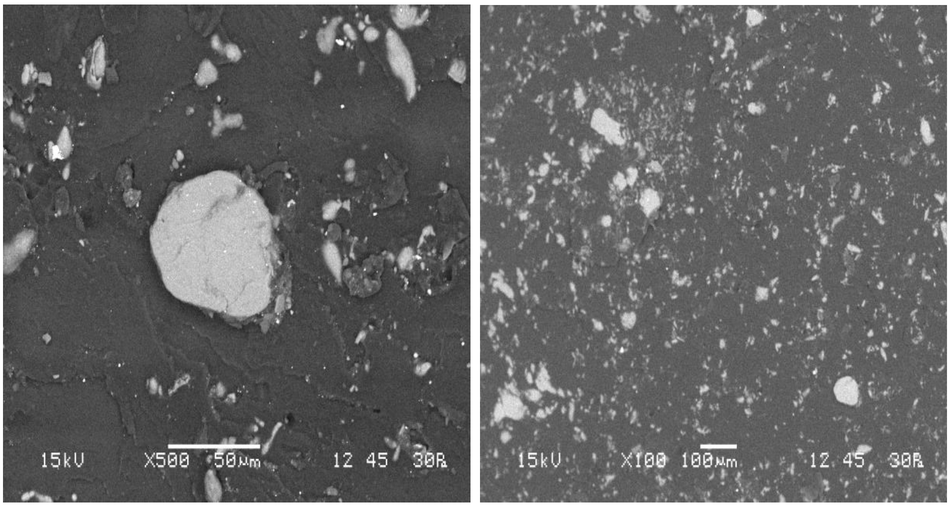


Figure 5.2 SEM images of pure red mud powder



(a) **(b)**

Figure 5.3 SEM images of (a) aluminium/epoxy (b) red mud/epoxy composites

The theoretically densities of fabricated composites are calculated by using rule of mixture [34]:

$$\rho_c = (1-\phi_f)\rho_p + \phi_f \rho_f \tag{5.1}$$

where ϕ_f is the volume fraction of filler, ρ_c, ρ_f, and ρ_p are the density of composite, filler material and matrix material, respectively.

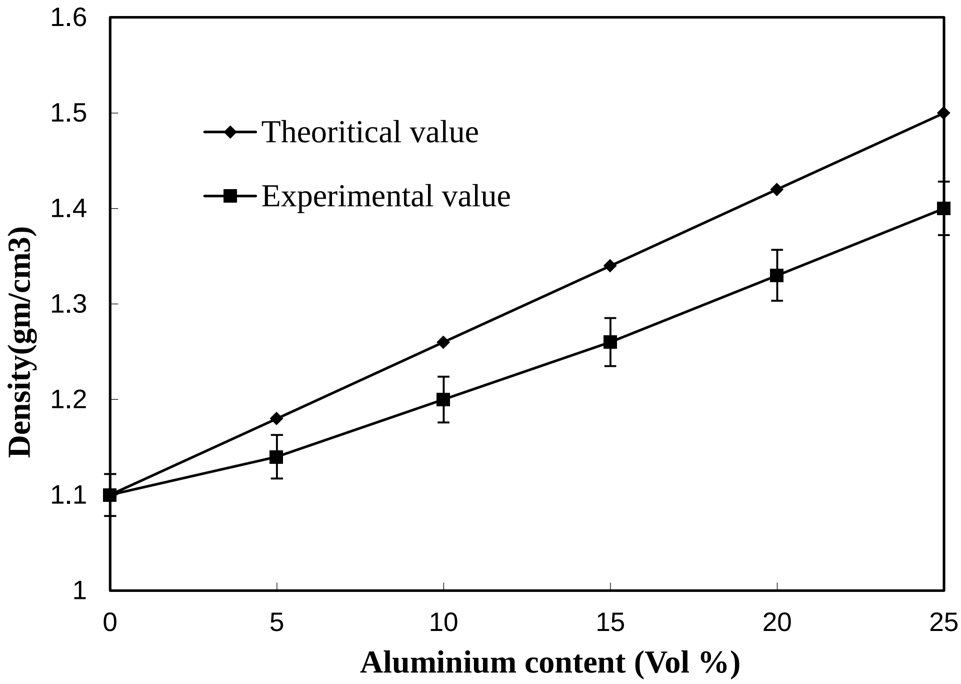


Figure 5.4 Effects in density with volume fraction of filler in aluminium/epoxy composites

The variation in the density of epoxy/aluminium composites with filler loading is shown in Figure 5.4. This Figure shows with the increases in volume fraction of filler material, the densities of epoxy/aluminium composites are also increases. Table 5.1 shows the void content in epoxy/aluminium composites for all the samples. With the increases of filler loading the voids content in composites also increases. It can also be seen that there are some differences between the experimentally and the theoretically densities of fabricated composites. This variation in density is due to the presence of voids and pores in fabricated composites.

Table 5.1 Variation in theoretical and experimental density with voids content in (Epoxy/aluminium) composites

Sr. No.	Filler content (vol%)	Density (g/cm^3) (Epoxy/aluminium)		Voids content (%)
		Theoretical	Experimental	
1	5	1.18	1.14	3.38
2	10	1.26	1.21	4.76
3	15	1.34	2.26	6.01
4	20	1.42	1.33	6.33
5	25	1.5	1.42	6.66

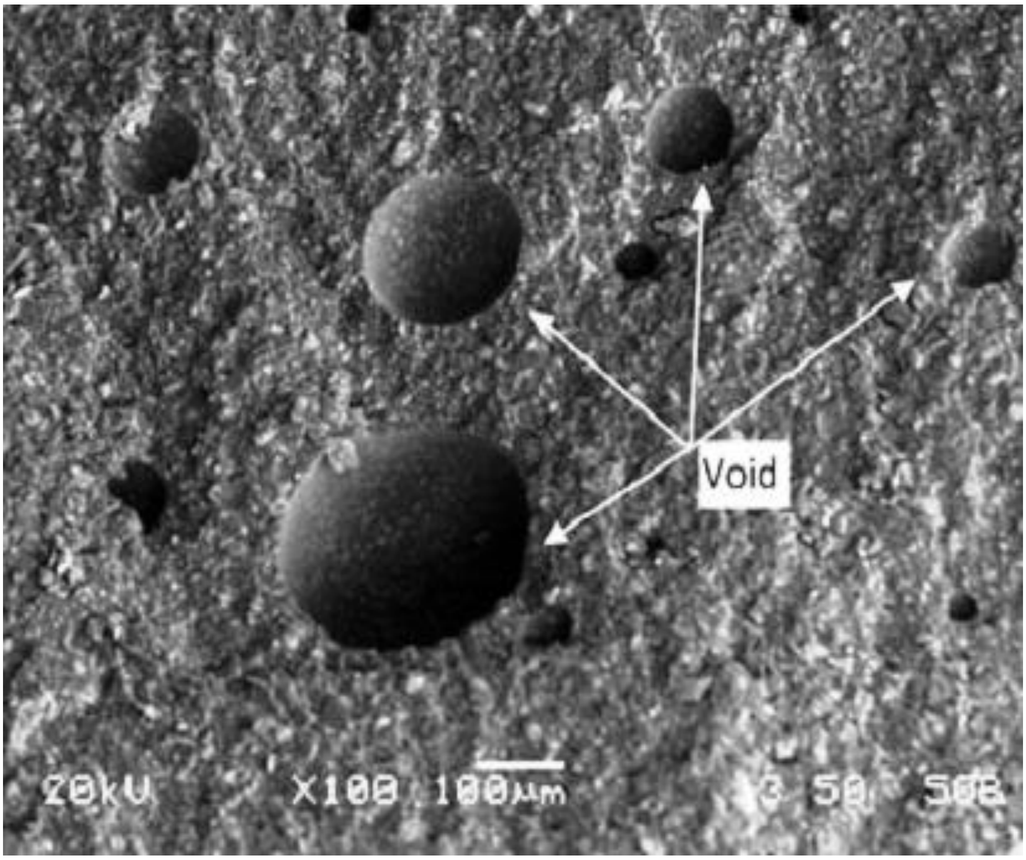


Figure 5.5 SEM to show the formation of voids during hand-lay-up technique.

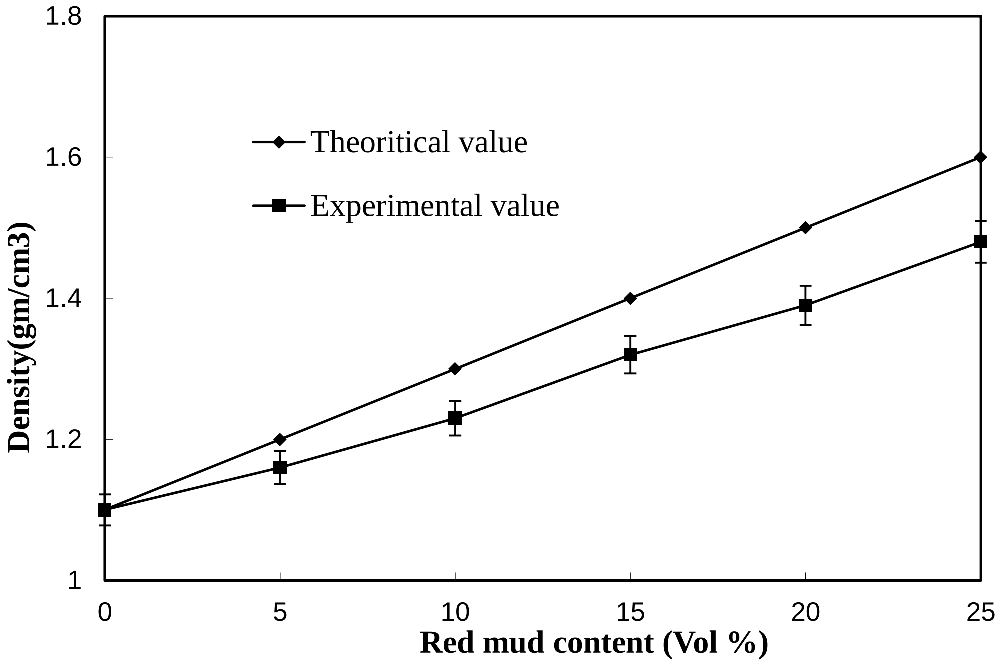


Figure 5.6 Effects in density with volume fraction of filler in Red mud/epoxy composites.

Table 5.2 Variation in theoretical and experimental density with voids content in (Epoxy/red mud) composites

Sr. No.	Filler content (vol%)	Density (g/cm^3) (Epoxy/red mud)		Voids content (%)
		Theoretical	Experimental	
1	5	1.20	1.16	3.33
2	10	1.30	1.23	5.38
3	15	1.40	1.32	5.71
4	20	1.50	1.39	7.33
5	25	1.60	1.48	7.50

Similar variation in density is noticed in the Epoxy/red mud composite. But the increment in densities of epoxy/red mud composites is slightly higher than epoxy/aluminium composites. The variation in densities of epoxy/red mud composites is shown in Figure 5.6 and its cross ponding calculated voids % is given in Table 5.2. With the increases of filler loading the voids content in composites also increases.

5.3 THERMAL CONDUCTIVITY OF FEBRICATED COMPOSITE SPECIMENS

5.3.1 Comparison between the values of k_{eff} obtained from various established theoretical model, experimental values and the proposed model.

Figure 5.7 shows the variation in the value of k_{eff} when micro-sized aluminium particles are added in epoxy matrix. It shows the comparison between the values obtained from various established theoretical model, experimental values and the proposed model. The theoretical calculated values for all the models and experimental measured values of k_{eff} for both set of fabricated composite i.e. epoxy/aluminium and epoxy/red mud composites are shown in Table 5.3 and Table 5.4 respectively.

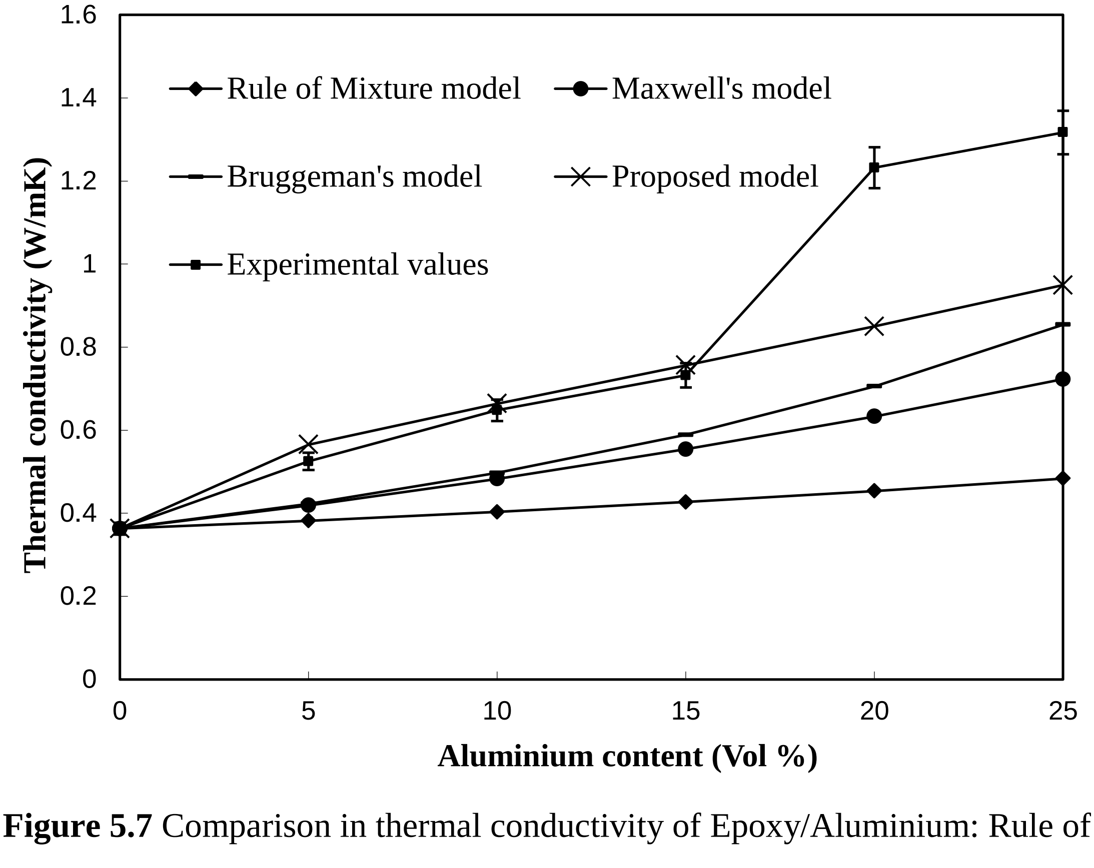


Figure 5.7 Comparison in thermal conductivity of Epoxy/Aluminium: Rule of mixture, Maxwell's model, Bruggman model, proposed model and Exp. values

Table 5.3 Theoretical calculated values of all models and experimental measured values of fabricated Epoxy/aluminium composites.

Sample no.	Content Aluminium Particles in vol%	K_{eff} of aluminium/epoxy polymer composites				
		Rule of mixture	Maxwell's Model	Bruggemen's Model	Proposed Model	Experimental
1	5	0.382	0.419	0.423	0.565	0.525
2	10	0.403	0.483	0.497	0.664	0.648
3	15	0.426	0.553	0.589	0.756	0.732
4	20	0.453	0.633	0.705	0.850	1.232
5	25	0.483	0.723	0.854	0.949	1.317

It is clear from the figure that there is significant increase in the value of k_{eff} as the content of aluminium particles is increasing. Also it is clear that while none of the established model are predicting the k_{eff} values correctly, only the model proposed by the authors are in close approximation with the measured values. Though this approximation is only up to 15 vol % after which a sudden jump in the value of measured k_{eff} is observed due to the formation of conductive chain for high filler loading. The vol % at which sudden rise in the value of k_{eff} is observed is called percolation threshold.

Similar behaviour is observed in case of epoxy-red mud composite as well, when red mud content increases beyond 20 vol % as shown in Figure 5.8. It is clear from both the figures that percolation threshold is not same for all combinations and is changes with either of the filler, matrix or both. The difference in the value of percolation threshold is mainly because of the difference in the value of intrinsic thermal conductivity of fillers, as red mud possesses less thermal conductivity when compared to aluminium.

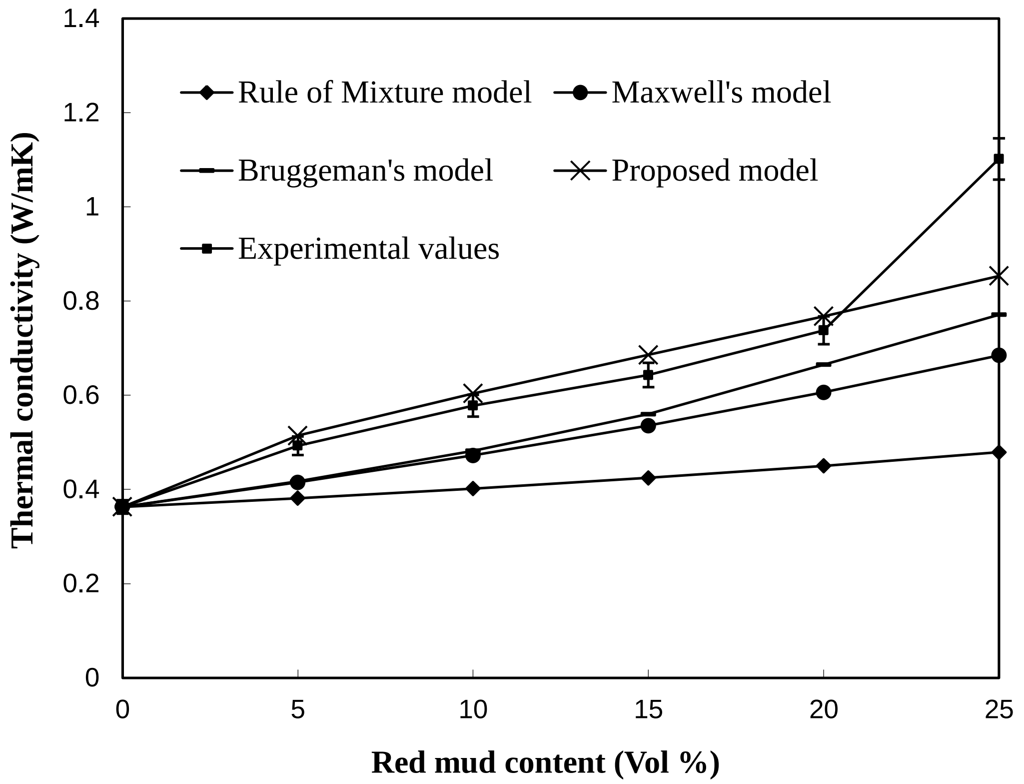


Figure 5.8 Comparison in thermal conductivity of Epoxy/red mud: Rule of mixture, Maxwell's model, Bruggman model, proposed model and Exp. values.

Table 5.4 Theoretical calculated values of all models and experimental measured values of fabricated Epoxy/red mud composites.

Sample no.	Content Red mud Particles in vol%	K_{eff} of red mud/epoxy polymer composites				
		Rule of mixture	Maxwell's Model	Bruggemen's Model	Proposed Model	Experimental
1	5	0.381	0.415	0.417	0.514	0.493
2	10	0.402	0.472	0.482	0.603	0.578
3	15	0.424	0.535	0.560	0.685	0.623
4	20	0.450	0.606	0.665	0.767	0.708
5	25	0.479	0.684	0.771	0.853	1.102

5.3.2 Variation between theoretical and experimental values of fabricated composites

Table 5.5 Theoretical and experimental value of k_{eff} of (Epoxy/aluminium) composites with error percentage.

Sr. No.	Filler content (vol%)	Thermal conductivity (W/m-K) (Epoxy/aluminium)		Associated Error (%)
		Theoretical	Experimental	
1	5	0.5655	0.525	7.70
2	10	0.6641	0.648	2.48
3	15	0.7562	0.732	3.30
4	20	0.85	1.232	31.00
5	25	0.9493	1.317	27.91

Table 5.6 Theoretical and experimental value of k_{eff} of (Epoxy/aluminium) composites with error percentage.

Sr. No.	Filler content (vol %)	Thermal conductivity (W/m-K) (Epoxy/aluminium)		Associated Error (%)
		Theoretical	Experimental	
1	5	0.5144	0.493	4.34
2	10	0.6037	0.578	4.44
3	15	0.6857	0.643	6.64
4	20	0.7678	0.738	4.03
5	25	0.8534	1.102	22.55

Table 5.5 and table 5.6 represent the variation between theoretical and experimental values of both set of fabricated composite specimens along with their associated errors. From the tables it is clear that the differences between the experimental and theoretical are well within the range of 10 % up to percolation threshold.

5.3.3 Comparison in thermal conductivity of both sets of fabricated composites based on proposed model and experimental measured values.

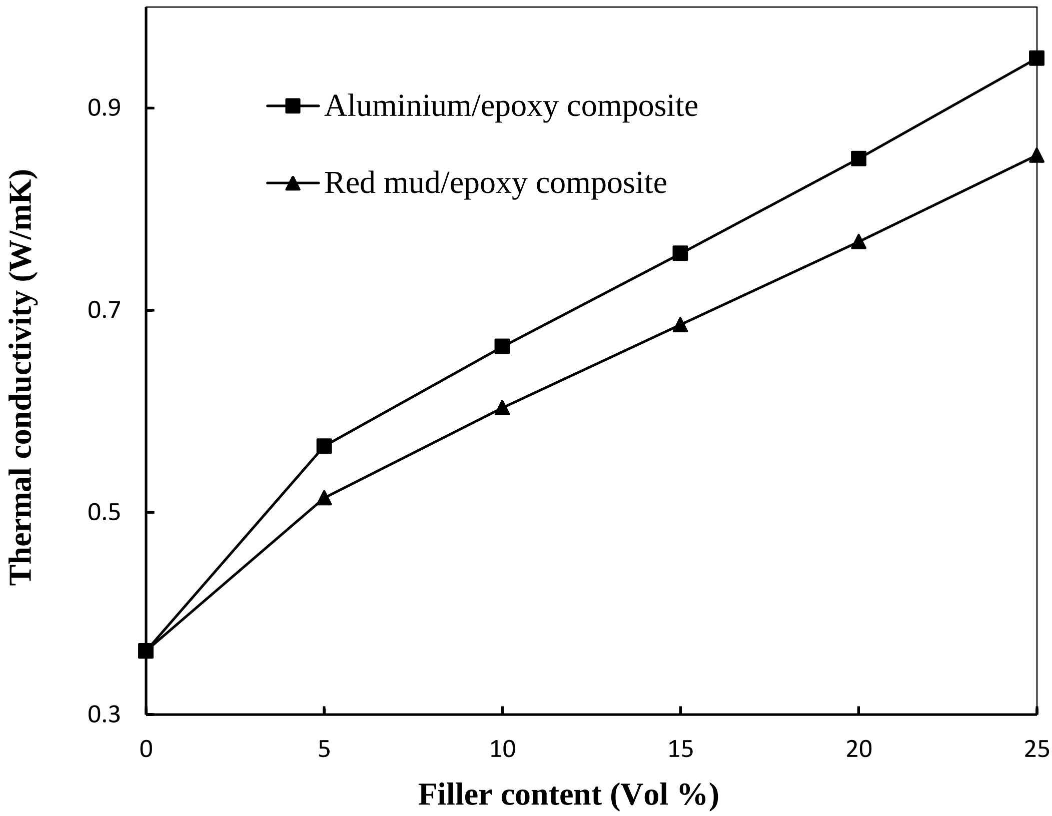


Figure 5.9 Variation of effective thermal conductivity with volume fraction of fillers: proposed model.

The variation in thermal conductivity of both sets of fabricated composite based on proposed model can be shown in 5.9. Similarly Figure 5.10 shows the comparison in thermal conductivity of both set of fabricated composites based on experimental values.

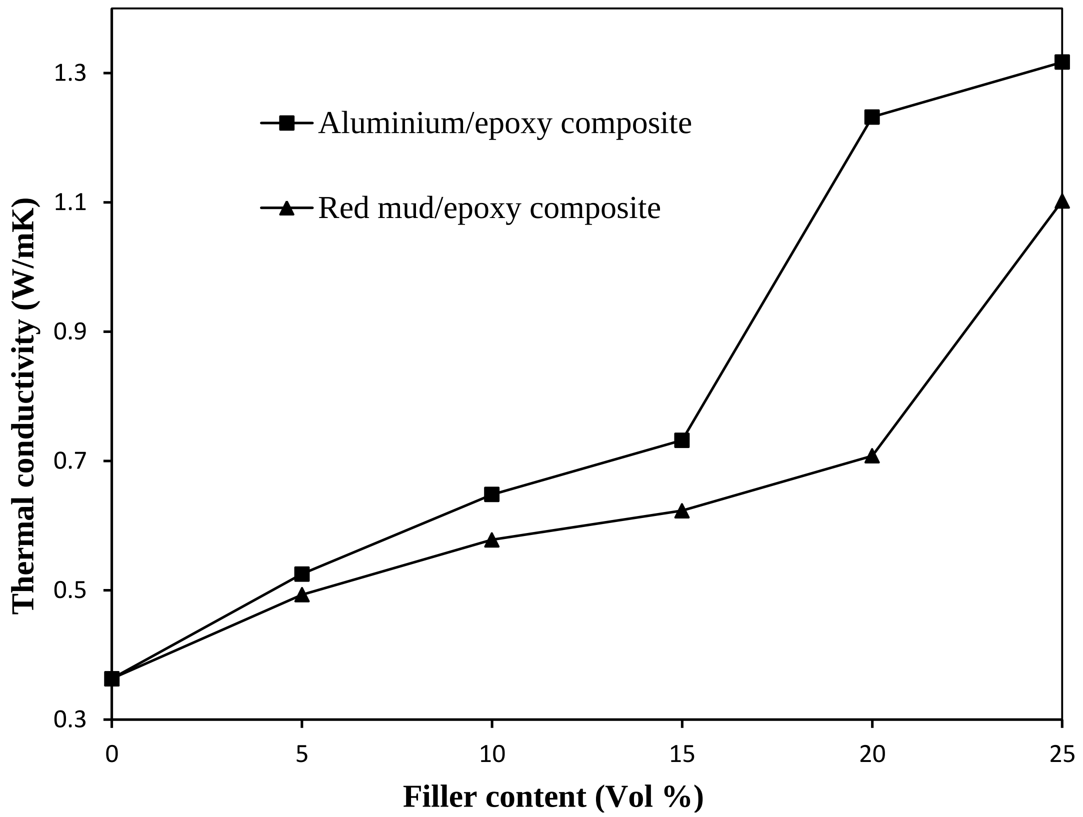


Figure 5.10 Variation of effective thermal conductivity with volume fraction of fillers: Experimental values.

It is also interesting to note that with addition of 25 vol % of aluminium particles, k_{eff} of epoxy increases to 160 % whereas same volume fraction of red mud increases the k_{eff} of epoxy by 135 %. It can be seen that though the difference between the intrinsic thermal conductivity of aluminium and red mud is huge but when they are reinforced in epoxy, the difference in k_{eff} of their composites is not much as it is explained by Bigg [48] that when the thermal conductivity of filler is 100 times more than that of matrix, there is not much increase in the value of k_{eff} is observed. So it can be seen that red mud, an industrial waste can replaces the costly aluminium metal.

Chapter Summary

This chapter has provided

- Surface morphology behaviour of both neat filler and fabricated composites.

- The effect of filler loading on density of polymer composites.
- The validation of proposed model through comparing all existing model and experimental values.
- Comparison of both set of polymer composites on the basis of theoretical and experimental values.

The next chapter gives the conclusion of research work on the basis of obtained results and also gives some ideas for future scope on this research work.

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CHAPTER 6

CONCLUSION AND FUTURE SCOPE

6.1 CONCLUSIONS

Based on the experimental and analytical work reported above, it can be concluded that:

1. Both set of composites i.e. epoxy/aluminium and epoxy/red mud composites can be successfully fabricated by simply hand lay-up technique.
2. The proposed mathematical model is predicting the k_{eff} values of both set of fabricated composites preicisely up to percolation threshold.
3. An improvement of about 161% in the value of thermal conductivity is recorded with addition of 25 vol % of aluminium filler in epoxy resin whereas when filler material is red mud it is noticed that k_{eff} increases 135% that of neat epoxy.
4. From the SEM images it is clear that particles are more or less elliptical in shape and are uniformly distributed in the matrix body.
5. There is large difference between the intrinsic thermal conductivity of aluminium and red mud powder, but it can be seen that there is only slight difference between k_{eff} of their composites. On that basis it can be siad that aluminium powder can be successfully replaced by an industrial waste i.e red mud.
6. These above fabricated composites can found its potential applications in electronics fields, printed circuit board, heat sink etc.

6.2 APPLICATIONS

With improved thermal conductivity the epoxy/aluminium and epoxy/red mud composites can be potential applications are follows:

1. Printed circuit board
2. Encapsulations
3. Heat Sink

4. electronic packaging

6.3 FUTURE SCOPES

1. These fillers can be fabricated with different matrix material.
2. Some other industrial wastes are also used as filler material for enhancing thermal conductivity of polymers.
3. A new mathematical model for evaluating effective thermal conductivity of polymer composites can be developed with parallel mode of heat conduction.
4. The effect of filler loading on strength, coefficient of expansion, dielectric behaviour of these fabricated composites can be investigated.

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LIST OF RESEARCH PUBLICATIONS

International Journal

1. **Johan Banjare**, Yagya Kumar Sahu, Alok Agrawal and Alok Satapathy. "Physical and thermal characterization of red mud reinforced epoxy composites: An experimental investigation" ***Procedia Materials Science (2014)***. (Accepted)

2. **Johan Banjare**, Yagya Kumar Sahu, Alok Agrawal and Alok Satapathy." Analytical and experimental approach to investigate effective thermal conductivity of particle reinforced polymer composites". ***Journal of Applied Polymer Science*** **(2014)**. (communicated)

International Conferences

1. **Johan Banjare**, Yagya Kumar Sahu, Alok Agrawal and Alok Satapathy. "Development of Mathematical Model to Evaluate Effective Thermal Conductivity of Particulate Filled Polymer Composites". ***International conference Emerging Materials and Processes- 2014, CSIR-IMMT Bhubaneswar.***

2. Yagya Kumar Sahu, **Johan Banjare**, Alok Agrawal and Alok Satapathy. "Establishment of an analytical model to predict effective thermal conductivity of fiber reinforced polymer composites". ***International Conference on Advancements in Polymeric Materials-2014, CIPET Bhubaneswar.***

3. Alok Agrawal, Yagya Kumar Sahu, **Johan Banjare** and Alok Satapathy. "Epoxy Composites Filled with Micro-sized Al_2O_3 Particles for Microelectronic Applications". ***International Conference on Functional Materials-2014, IIT Kharagpur.***